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XVIII.

CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF
HARVARD COLLEGE.

THE VOLUMETRIC DETERMINATION OF COMBINED
NITROUS ACID.

BY LEONARD P. KINNICUTT AND JOHN U. NEF.

Presented May 9th, 1883.

THE amount of nitrous acid contained in commercial samples of potassic and sodic nitrites has been commonly determined in the following manner. The nitrites are dissolved in slightly acidulated water; a solution of potassic permanganate is added till the oxidation of the nitrous acid is nearly completed; the solution is then made strongly acid, and potassic permanganate added until the solution has a faint red color.

This method is far from satisfactory, closely agreeing results being rather the exception than the rule. The cause is most probably due to the escape of a small amount of nitrous acid, and also to the slow oxidation of the last traces of the nitrous acid by the potassic permanganate.

A further study of the analyses of nitrites volumetrically seemed desirable, and after numerous experiments the following process, or modification of the old process, as it might more properly be called, was devised.

The sample of nitrite is dissolved in cold water, one part of the salt to at least three hundred parts of water. To this solution a deci-normal solution of potassic permanganate is added, drop by drop, till the liquid has a decided and permanent red color, then two or three drops of dilute sulphuric acid, and immediately afterwards an excess of the potassic permanganate solution. The liquid, which should now be of a dark red color, is made strongly acid with sulphuric acid, heated to boiling, and the excess of potassic permanganate determined by means of a deci-normal solution of oxalic acid.

Analyses of samples of both potassic and sodic nitrites, made in accordance with the above modification, show that results agreeing very closely with one another can easily be obtained.

Potassic Nitrite Solution.

[One litre contained 4.3550 grammes.]

Taken.	KNO_2 found.
25 c.c. = 0.1089 gramme	0.09194 gramme = 84.44 %
35 c.c. = 0.1525 "	0.1289 " 84.48 %
40 c.c. = 0.1742 "	0.1472 " 84.51 %
55 c.c. = 0.2395 "	0.2024 " 84.49 %
60 c.c. = 0.2613 "	0.2210 " 84.53 %

Sodic Nitrite Solution.

[One litre contained 3.090 grammes.]

Taken.	NaNO_2 found.
25 c.c. = 0.0919 gramme	0.07713 gramme = 83.95 %
35 c.c. = 0.1287 "	0.1082 " 84.07 %
45 c.c. = 0.1654 "	0.1390 " 84.05 %
53 c.c. = 0.1949 "	0.1639 " 84.03 %
60 c.c. = 0.2206 "	0.1854 " 84.05 %

The volumetric method for the determination of sulphites is also most unsatisfactory. The study of this subject was therefore given to Mr. R. Penrose, a student in this laboratory. He experimented both with potassic permanganate and oxalic acid as above, and also with iron alum and the permanganate; but, although many variations of these processes were tried, and the work carefully conducted, the results were unsatisfactory.